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PATENT ABSTRACTS OF JAPAN

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(71)Applicant : APPLIED MATERIALS INC

(72)Inventor : GUPTA ANAND
TIRUNELVELI S RAVI

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(54) PLASMA FLUORINE-RESISTANT ALUMINA CERAMIC MATERIAL LOW IN GENERATION OF PARTICLE AND ITS PRODUCTION

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a polycrystallin material showing high resistance to plasma fluorine and reduction in generation of particles.

SOLUTION: This polycrystallin alumina ceramic material is produced by a step for forming a non-sintered compact having alumina and a binder and a step for sintering the non-sintered compact for about 8-12 hours. An area % of the non-sintered particle in a polycrystalline alumina ceramic material is preferably <0.1 area %. Consequently the generation of particles after exposure to plasma fluorine is reduced.

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CLAIMS

[Claim(s)]
[Claim 1] Technique equipped with the step which forms the non-sintered compact which is the technique of manufacturing the polycrystal alumina ceramic material which has resistance, and equips a plasma fluorine with (i) alumina and a binder, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact.
[Claim 2] Technique according to claim 1 area % of the non-sintered grain of the aforementioned polycrystal alumina ceramic material is below about 0.1 area %.
[Claim 3] Technique according to claim 2 area % of the non-sintered grain of the aforementioned polycrystal alumina ceramic material is about 0.01 area % - about 0.1 area %.
[Claim 4] Technique according to claim 1 the step described above, iied) sintered is a step which performs non-energized sintering.
[Claim 5] Technique according to claim 1 by which the step described above, iied) sintered is executed at the temperature of about 1400 degrees C - 1700 degrees C.
[Claim 6] Technique according to claim 5 by which the step described above, iied) sintered is executed at the temperature of about 1600 degrees C - 1650 degrees C.
[Claim 7] Technique [equipped with the material as which the aforementioned binder is chosen from the group which consists of a silica, a calcium oxide, magnesium oxides, and such mixture] according to claim 1.
[Claim 8] Technique according to claim 1 that the aforementioned sheep sintered compact is equipped [an alumina] with about 0.7 wts% - 0.3wt% for about 99.3 wts% - 99.7wt% and the aforementioned binder.
[Claim 9] It sets to the step described above, iied) sintered, and, for the aforementioned sheep sintered compact, a density is 3 at least 3.8g/cm. Technique according to claim 1 sintered so that it may become.
[Claim 10] Technique equipped with the step which sinters the aforementioned sheep sintered compact during the step which forms the non-sintered compact which is the technique of manufacturing the polycrystal alumina ceramic material which has resistance, and equips a plasma fluorine with (i) alumina and a binder, and the time when area % of the non-sintered grain of the (ii) aforementioned alumina ceramic material does not exceed 0.1 area % at, and becomes.
[Claim 11] Technique equipped with the step which forms the non-sintered compact which is the technique of manufacturing the polycrystal alumina ceramic material which has resistance in a plasma fluorine, and is equipped with about 0.7 wts% - 0.3wt% for the binder chosen from the group which consists (i) alumina of about 99.3 wts% - 99.7wt%, a silica, a calcium oxide, magnesium oxides, and such mixture, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact.
[Claim 12] It is the technique of manufacturing the polycrystal alumina ceramic material which has resistance in a plasma fluorine. (i) alumina About 99.3 wts% - 99.7wt%, The step which forms the non-sintered compact equipped with about 0.7 wts% - 0.3wt% for the binder chosen from the group which consists of a silica, a calcium oxide, magnesium oxides, and such mixture, (ii) Technique have the step

to which about 8 - 12 hours sinters the aforementioned sheep sintered compact, and area % of the non-sintered grain of the aforementioned alumina ceramic material does not exceed 0.1 area %.

[Claim 13] It is the technique of manufacturing the polycrystal alumina ceramic material which has resistance in a plasma fluorine. (i) alumina About 99.3 wts% - 99.7wt%, The step which forms the non-sintered compact equipped with about 0.7 wts% - 0.3wt% for the binder chosen from the group which consists of a silica, a calcium oxide, magnesium oxides, and such mixture, (ii) Technique have the step to which about 8 - 12 hours sinters the aforementioned sheep sintered compact at the temperature of about 1400 degrees C - 1700 degrees C, and area % of the non-sintered grain of the aforementioned alumina ceramic material does not exceed 0.1 area %.

[Claim 14] Technique according to claim 13 by which the step described above, iied) sintered is executed at the temperature of about 1600 degrees C - 1650 degrees C.

[Claim 15] The ceramic material manufactured by the technique of manufacturing the polycrystal alumina ceramic material equipped with the step which forms the non-sintered compact which is a ceramic material and is equipped with (i) alumina and a binder, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact which has resistance in a plasma fluorine.

[Claim 16] The ceramic material which is equipped with the step which forms the non-sintered compact which is a ceramic material and is equipped with (i) alumina and a binder, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact, and is manufactured by the method of manufacturing the polycrystal alumina ******** material which has resistance in a plasma fluorine that area % of the non-sintered grain of the aforementioned polycrystal alumina ceramic material is below about 0.1 area %.

[Claim 17] The ceramic material which is equipped with the step which forms the non-sintered compact which is a ceramic material and is equipped with (i) alumina and a binder, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact, and is manufactured by the method of manufacturing the polycrystal alumina ceramic material which has resistance in a plasma fluorine that area % of the non-sintered grain of the aforementioned polycrystal alumina ceramic material is about 0.01 area % - about 0.1 area %.

[Claim 18] It is a ceramic material. (i) alumina About 99.3 wts% - 99.7wt%, The step which forms the non-sintered compact equipped with about 0.7 wts% - 0.3wt% for the binder chosen from the group which consists of a silica, a calcium oxide, magnesium oxides, and such mixture, (ii) Ceramic material manufactured by the technique of manufacturing the polycrystal alumina ceramic material equipped with the step to which about 8 - 12 hours sinters the aforementioned sheep sintered compact which has resistance in a plasma fluorine.

[Claim 19] It is a ceramic material. (i) alumina About 99.3 wts% - 99.7wt%, The step which forms the non-sintered compact equipped with about 0.7 wts% - 0.3wt% for the binder chosen from the group which consists of a silica, a calcium oxide, magnesium oxides, and such mixture, (ii) Ceramic material manufactured from the method of manufacturing the polycrystal alumina ceramic material which has resistance in a plasma fluorine that have the step to which about 8 - 12 hours sinters the aforementioned sheep sintered compact, and area % of the non-sintered grain of the aforementioned alumina ceramic material does not exceed 0.1 area %.

[Claim 20] It is a ceramic material. (i) alumina About 99.3 wts% - 99.7wt%, The step which forms the non-sintered compact equipped with about 0.7 wts% - 0.3wt% for the binder chosen from the group which consists of a silica, a calcium oxide, magnesium oxides, and such mixture, (ii) It has the step to which about 8 - 12 hours sinters the aforementioned sheep sintered compact at the temperature of about 1400 degrees C - 1700 degrees C. The ceramic material manufactured by the method of manufacturing the polycrystal alumina ceramic material which has resistance in a plasma fluorine that area % of the non-sintered grain of the aforementioned alumina ceramic material does not exceed 0.1 area %.

[Claim 21] The production which equips a plasma fluorine equipped with the step which forms the non-sintered compact which is a production and is equipped with (i) alumina and a binder, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact with the ceramic material manufactured by the technique of manufacturing the polycrystal alumina ceramic material

which has resistance.
[Claim 22] The production according to claim 21 which are parts for using for a vacuum processing chamber.
[Claim 23] The production according to claim 22 chosen from the group which consists of a gas inspersion shield, a chuck, a nozzle, a susceptor, a heater plate, a clamping ring, a wafer boat, and a chamber wall.
[Claim 24] The production according to claim 23 which is a gas inspersion shield.
[Claim 25] A vacuum devices equipped with the production which are parts for using for the vacuum processing chamber equipped with the step which forms the non-sintered compact which is a vacuum processor and is equipped with (i) alumina and a binder, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact which equips a plasma fluorine with the ceramic material manufactured by the technique of manufacturing the polycrystal alumina ceramic material which has resistance.
[Claim 26] A chemistry gaseous-phase deposition reactor equipped with the gas inspersion shield equipped with the step which forms the non-sintered compact which is a chemistry gaseous-phase deposition reactor and is equipped with (i) alumina and a binder, and the step to which about 8 - 12 hours sinters the (ii) aforementioned sheep sintered compact which equips a plasma fluorine with the ceramic material manufactured by the technique of manufacturing the polycrystal alumina ceramic material which has resistance.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] this invention has high resistance in etching by the fluorine plasma, and relates to the improved alumina ceramic material which is characterized by reducing grain occurrence. this invention relates to the production (article of manufacture) further equipped with this improved ceramic material about the technique for making this improved ceramic material.
[0002]
[Description of the Prior Art] Typically, the alumina ceramic material of polycrystal nature is produced according to the following sintering processes. The pulverization alumina which has desired particle size distribution (it has an about 1 micrometer - 3 micrometers pitch diameter typically) is mixed with a binder, presses the mixture of this alumina and a binder, and forms a non-sintered compact (green body) (green body). Generally, this non-sintered compact has the composition which has alumina 99.5%, a silica as a binder, and the mixture of MgO and CaO 0.5%. Then, a non-sintered compact is sintered and, typically, is performed in temperature of about 1650 degrees C in the air of an ordinary pressure (non-energized sintering) for about 4 hours.
[0003] Grain grows in the midst of a sintering process. For example, in a general sintering process, a distribution of the sintering grain size in which mean grain size has the domain of about 1-30 micrometers by about 6 micrometers is given. Here, these particle size distribution are ASTM (American Society ofTesting and Materials) specification E. 1181-87 (determination of duplex grainsizes) And E 112-88 (determination of average grain sizes) It determines by the well-known technique, such as indicated technique. When alumina grain grows, transition of a binder phase happens. It moves to the field where smaller grain exists, and the transferred binder phase encloses this small grain. silica (J. -- W. -- Welch, Nature, vol.186, 546 or less p, and (1960) -- reference) which is the principal component of the binder phase with a typical alumina Since it is very hard to carry out a mixing, the surrounded binder phase checks that the small grain which isolates and exists grows further. Probably, such non-sintered grain which isolates and exists is in the domain of about 0.1-0.5-micrometer diameter. Typically, it will remain, without sintering about 1% of alumina grain.
[0004] Even if it has the property of requests, such as a high intensity and a fracture toughness, the known polycrystal alumina ceramic material is not proved to the intended use as which plasma exposure is required, if the resistance over a specific fluorine plasma is enough. A known alumina tends to receive etching by the **** fluorine plasma generated in a chamber cleaning process in a chemistry gaseous-phase deposition (CVD) reactor. In this process, the dielectric layer residue deposited on the chamber of a reactor is removed using the plasma fluorine (CF4 : for example, NF3 plasma, CF4 :O2 plasma, and an N2 O plasma) isolated from the fluorine inclusion gas of fluoridation carbon or others.
[0005] ; which an alumina receives to a plasma fluorine in itself, and has high resistance, therefore the sapphire which is the alumina of a pure single crystal are one of the matter most known as matter with which etching progresses late.

[0006] Etching of a polycrystal alumina ceramic material is mainly produced in a binder phase. As a result of being etched in a binder phase, the small grain which was not sintered separates. This grain that separated may be emitted from the front face of a ceramic material. When the alumina ceramic material of such a polycrystal is used for the environment where it is exposed to the plasma fluorine of the inside of CVD reactor, or others, this serves as a pollution source.
[0007] Change from the beginning a distribution of using [ceramic materials other than a using / the binder with little influence to a ****ing / in :polycrystal alumina ceramic in which the following things are contained, make the rate of an alumina high, for example, / to binder 0.01% /-alumina 99.9%; fluorine plasma /; alumina];, and the alumina grain size of a non-sintered compact into the solution considered to the problem of grain occurrence.
[0008] the intended use from which especially grain occurrence poses a problem -- processing of the semiconductor wafer in a chemistry gaseous-phase deposition (CVD) system -- it is -- for example, Chang ** -- it is the U.S. patent-application S.N."5000" equipment of applied ************ indicated by No. 08/136,529 In drawings 1 and 2, CVD reactor for an explanation of the conventional technique is illustrated. The CVD system 10 is equipped with the deposition chamber 12, the vacuum channel 13, the evacuation system 14, the gas inflow means 16, the gas inspersion shield (gas distribution shield) 17, the blocker 18, the wafer lift 20, the baffle plate 22, the lift finger 24, and the susceptor lift 26 as shown in drawing 1 . A substrate 28 is a semiconductor wafer etc. and is arranged on a susceptor 30. The heating means 32 is the array of the 1000W lamp in the exterior which has turned the light which it collimated in the orientation which lets the quartz watch window 36 pass, for example, and keeps processing temperature uniform. Deposition or the reaction zone 34 exists in the information on a substrate.
[0009] The gas inspersion shields 17 are the flat annular parts surrounding the surroundings of a blocker 18, and as shown in drawing 2 , they are being fixed so that it can remove to the chamber lid 38 with two or more aluminum clips 40. Typically, the gas inspersion shield 17 is equipped with the polycrystal alumina ceramic material.
[0010] In the typical deposition process performed in CVD system illustrated here, process gas (namely, reactant gas and carrier gas) advances into the deposition chamber 12 through the blocker 18 of the gas inflow means 16 and a "shower head" type. The blocker 18 has much openings covering the area according to the area of the substrate 28 under it. The spacing of a blocker 18 and the substrate 28 can be adjusted to about 200 to 1000 mils (5-25mm), and has demarcated the reaction field 34. A deposition reaction is executed and gas is purged from a chamber 12. It is NF3 whenever a wafer is processed. Or C2 F6 / NF3 / O2 A chamber is cleaned using cleaning gas, such as a gas mixture object.
[0011]
[Problem(s) to be Solved by the Invention] However, when the gas inspersion shield 17 is equipped with a polycrystal alumina ceramic material, a shield receives etching by above-mentioned cleaning gas or an above-mentioned gas mixture object, and, as a result, grain generates it. The silicon wafer by which the grain which has the size of about 0.2-0.5 micrometers may occur, and this is processed by the CVD system may be polluted. In the front face of a silicon wafer after processing 100 silicon wafers, it is 200/cm2. Or the total of the grain beyond it may be observed. The total of this grain is the high thing which cannot be received.
[0012] The need of improving a polycrystal alumina ceramic material, and the need for the technique of manufacturing the material exist continuously. This material should show the high resistance over a plasma fluorine, and should show a reduction of grain occurrence especially. It is required to equip the gas inspersion shield for using for a CVD system especially with such an improved material.
[0013]
[Means for Solving the Problem] According to one characteristic feature of this invention, a plasma fluorine equipped with the step which forms the non-sintered compact equipped with the alumina and the binder, and the step which sinters this non-sintered compact for about 8 hours to 12 hours is provided with the method of manufacturing the polycrystal alumina ceramic material which has resistance.
[0014] A plasma fluorine equipped with time when area % of the step which forms the non-sintered

compact equipped with the alumina and the binder according to another characteristic feature of this invention, and the grain which is not sintered in an alumina ceramic material does not exceed 0.1 area % at, and becomes, and the step which sinters this non-sintered compact is provided with the method of manufacturing the polycrystal alumina ceramic material which has resistance.
[0015] According to another characteristic feature of this invention, the production which the ceramic material manufactured according to above-mentioned technique is offered, and was equipped with the above-mentioned ceramic material is offered again. The parts useful to vacuum processors, such as CVD chamber, in a desirable example, especially gas inspersion shield (gas distribution shield) of a production of this invention It is contained.
[0016] Other purposes, characteristic features, and advantages of this invention will become clear from the following detailed explanations at this contractor. However, although the gestalt and the specific example of implementation of the following invention suggest the desirable example of this invention, they are not indicated for instantiation and are not for limitation. Without leaving the essence of this invention, various change and deformation may be made in the domain of this invention, and this invention contains such all deformation.
[0017]
[Embodiments of the Invention] In order to make the alumina ceramic material which has high resistance to a fluorine plasma, we found out that it was not necessary to change the sintering process factor of others, such as composition, sintering temperature, etc. of a non-sintered compact which can affect the mechanism of sintering, that what is necessary is just to extend the time of sintering operation of an alumina sheep sintered compact (alumina green body) from 4 conventional hours in 8 - 12 hours.
[0018] To the alumina ceramic material which can lower 1 figure of the percentages of non-sintered grain, namely, is known from the former, the place which was 1 area % can be made now below into about 0.1 area % by lengthening sintering time to predetermined sintering temperature and composition of a non-sintered compact. The amount of the grain emitted from the ceramic material exposed to a plasma fluorine also decreases corresponding to this.
[0019] The non-sintered compact for manufacturing a polycrystal alumina ceramic material by sintering according to this invention may be formed from the alumina fine particles which have composition of all requests. Desirable composition of fine particles has about 0.7 - 0.3% of the binders chosen from the group which changes from a silica, a calcium oxide, magnesium oxides, and those mixture to 99.3% - 99.7% of alumina abbreviation. This fine-particles constituent may have the grain-size distribution which exists from the former.
[0020] The selected alumina fine particles are fabricated by the non-sintered compact by the means of common knowledge [former]. Preferably, the domains of the pressure used for a manufacture of a non-sintered compact are about 5, 000psia-14, and 000psia, and are about 7, 000psia-10, and 000psia still preferably. The initial density of a non-sintered compact is in the domain of about 1.8-2.2g/cm preferably.
[0021] Then, a non-sintered compact is sintered for about 8 to 12 hours. if sintering time exceeds 12 hours, to the direction which does not desire the structure of a non-sintered compact, the crystal nucleation of-like secondary phase will arise and it will change [it will be alike and] When sintering is shorter than 8 hours, not coming to decrease the total of non-sintered grain to a desirable grade, and reducing occurrence of grain corresponding to this is not realized. Because of the purpose of the economical efficiency of a process, about 8 - 10 sintering hours are desirable. At this time, the density after **** is 3 at least 3.8g/cm. It is desirable that **** is performed so that it may become.
[0022] A sintering process here has the desirable non-energized process performed in air. Moreover, a sintering process may be performed in some sintering conditions from the former of others, such as the inert gas ambient atmosphere. About 1400 degrees C - 1700 degrees C of sintering processes may be preferably performed at the temperature of about 1600 degrees C - 1650 degrees C still preferably. Here, in the enhancement sintering technique of this invention, that a required thing changes only the length of sintering time only emphasizes things. The parameter of other sintering processes is good, not changed.
[0023] In the alumina ceramic material which is manufactured according to the process of this invention

and which has resistance in a plasma fluorine, it is shown that the amount of non-sintered grain is reduced remarkably so that it may be proved by comparison of the material illustrated in drawings 3 and 4.

[0024] The amount of non-sintered grain can be determined according to the technique learned in the conventional technique. One of the typical technique examines the microphotography of the field chosen at random [two or more alumina ceramic materials], and presence of non-sintered grain is determined by check. It is the whole surface product to which the area of non-sintered grain was determined and the alumina ceramic material carried out microscope photography of the whole area of non-sintered grain, and is ****. This quotient is area % and gives the scale of the amount of non-sintered grain.

[0025] The percentage of non-sintered grain shows below about 0.1 area %, and, as for the alumina ceramic material manufactured according to the process of this invention, about 0.01 area % - 0.1 area % is shown preferably.

[0026] The grain which is exposed to a plasma fluorine and occurs from the alumina ceramic material of this invention also decreases according to this. For example, the grain contamination of a silicon wafer generated by move of the grain produced after exposing the parts of a CVD system equipped with an alumina ceramic material to a fluorine plasma can be quantified using a standard technique. Furthermore, specifically, the grain occurrence from an alumina ceramic material can be determined by measuring the total of the grain on the front face of a wafer in the deposition step performed after CVD chamber cleaning.

[0027] If a typical standard technique is followed, the total of grain is U.S. California Mountain View. It is commercially available from Tencor Instruments Inc. Tencor Surfscan 6200 It uses and determines. Tencor Surfscan 6200 By measuring the amount of the light (given by 30mW Ar-ion laser with a wavelength of 488nm) scattered about by grain, the number of the grain on a front face is determined. Tencor Surfscan6200 About a principle of operation and the equipment of a relation, it is Surface Contamination Detection. : AnIntroduction (R.Johnson, Ed., Tencor Instruments Inc, MountainView, and CA 1990) It is explained. For the decision of the particle number on the front face of a material, you may use the other known technique.

[0028] The grain occurrence from the alumina ceramic material manufactured according to the process of this invention is remarkably reduced compared with the conventional process equivalent to this, i.e., the alumina ceramic material with which sintering time was manufactured in about 1 - 4 hours according to the process that other parameters and composition of a non-sintered compact are the same. A decrement of occurrence of the grain in comparison with the ceramic material typically manufactured according to a process conventionally [above-mentioned / considerable] is it that it is desirable and is few about 60% - 90% very preferably about 60% about 50% at least, when an above-mentioned desirable procedure determines.

[0029] The alumina ceramic material which has deposition is given to the plasma fluorine [the process of this invention] which can be used for a manufacture of various productions, and this production is for using for the environment where it is exposed to a plasma fluorine, preferably. The parts used for a bell jar, a crucible, and other vacuum processors are contained in such a production. The parts with which the production in the domain of this invention is still specifically used for vacuum processors, such as a gas inspersion shield, a chuck, a nozzle, a susceptor, a heater plate, a clamping ring, a wafer boat, or a chamber wall, are contained. Such a production may have one or more front faces which have the whole or coating which is partially formed with the ceramic material of this invention, or is equipped with the ceramic material of this invention.

[0030] If the gas inspersion shield has the alumina ceramic material of this invention which is explained here, there are especially profits. If a gas inspersion shield is such, it will be enabled to use for various known equipments for processing of the semiconductor material of a silicon wafer or others. the CVD system given to the known equipment which can use the above-mentioned gas inspersion shield advantageously by applied ************ of U.S. California Santa Clara -- it is -- for example, Chang ** -- the equipment indicated by U.S. patent-application S.N.08 / No. 136,529 or Tseng ** -- the equipment indicated by U.S. patent S.N.08 / No. 314,161 is included Furthermore, as a concrete

example, it is a precision 5000CVD reactor. (Precision 5000 CVD reactor) (commercially available from applied ************) It is mentioned.
[0031]
[Example] The example of limitation which is not a sake is shown below and this invention is further illustrated with reference to this.
[0032] Example 1 alumina fine particles (commercially available from Alcoa) are mixed with the silica of fine particles, and MgO and CaO, and the constituent equipped with MgO0.15wt% and CaO0.15wt% was obtained silica 0.2wt% alumina 99.5wt% (weight %). This constituent was covered over the ball mill and the mean particle diameter was set to 0.2 micrometers. and this constituent -- compressing -- the diameter of about 13 inches -- inner -- the diameter of a hole -- about 6 inches and thickness -- 0.25 inches and a density -- 3g/cm3 it is -- the non-sintered compact of the configuration of a gas inspersion shield was formed
[0033] In air, the sintering time under 1650 degrees C and on a title is 8 hours (1650 degrees C was actually maintained for 8 hours), and non-energized sintering of the non-sintered compact according to this invention was carried out.
[0034] And occurrence of grain was measured whenever it sintered the gas inspersion shield. Each shield sintered and made is the reactor in which it was installed in the precision 5000CVD reactor, and this shield was installed, and processed the silicon wafer with a diameter of 150mm (6 inches).
[0035] Si3 N4 from the former which performs chamber cleaning The chemistry gaseous-phase deposition process was executed by CVD reactor as the following. The silicon wafer was introduced in the vacuum deposition chamber of CVD reactor, and it was heated by 400 degrees C. SiH4 (180sccm), N2, and (1800sccm) NH3 (75sccm) were introduced in the chamber, and the chamber pressure was stabilized by 4.5 tolls. And the 450W plasma was lit into the chamber and deposition operation was executed for 1 minute. Then, the chamber was exhausted with the pump until it became the base pressure (100mm toll), and the wafer was taken out.
[0036] After taking out a wafer, the inside of a chamber was cleaned using the plasma cleaning process. CF4 and (1500sccm) N2 O (750sccm) were introduced in the chamber, the pressure was stabilized by five tolls, and the 750W plasma was lit into the chamber. Plasma cleaning was performed for 30 seconds. Then, the inside of a chamber was exhausted with the pump until it became the base pressure.
[0037] Next, it is Si3 N4 within a chamber. By depositing, seasoning of the chamber was carried out for 15 seconds. The step of this seasoning was executed by the same technique as the step of above-mentioned deposition.
[0038] In the procedure of the 1st experiment, the step of seasoning was performed, whenever the cleaning cycle was completed. The step of seasoning was excepted in the procedure of the 2nd experiment.
[0039] By the technique the number of the grain which has the size of 0.2 micrometers or more on the front face of a silicon wafer is explained to be here before and behind CVD deposition process Tencor Surfscan 6200 Counting was carried out using the wafer surface scanner.
[0040] The result is shown in the following table 1.
[0041]
[Table 1]

８時間焼結後のシールド	Ｓｉウエハ上の粒子総数	
	シーズニングあり	シーズニングなし
ウエハ５枚処理後	25-30	25-30
ウエハ２００枚処理後	25-30	25-30

Example 2 - In the comparison this example with a known process, although the non-sintered compact was formed from the alumina covered over the above-mentioned ball mill, the sintering is performed according to a known process, namely, the sintering time on a title is 4 hours (it actually maintains at

1650 degrees C for 4 hours), and it manufactured the gas inspersion shield for a comparison. Other process conditions were the same in having used in the example 1.
[0042] And each shield for this comparison was measured by the technique completely same about grain occurrence as an example 1. The result shown in following Table 2 was found out.
[0043]
[Table 2]

４時間焼結後のシールド	Ｓｉウエハ上の粒子総数	
	シーズニングあり	シーズニングなし
ウエハ５枚処理後	7	35
ウエハ２００枚処理後	35	400

If it compares with the result of an example 1, it will become clear that it has been the characteristic feature that occurrence of the grain according [the shield manufactured according to the technique of this invention] to exposure to a plasma fluorine is improved remarkably. Even if there is little occurrence of the grain from the shield of this invention and it performs the cycle of wafer processing repeatedly on a number, it is substantially fixed.
[0044] A reduction of the grain occurrence realized by this invention is especially remarkable in comparison with the known method of not having the step of seasoning. The grain which occurs after the shield for a comparison performs a wafer processing cycle 200 times has more no less than 800% than the case where it sees after performing the number-of-times wafer processing cycle with the same shield of this invention.
[0045] The material of this invention conquers the difficulty related to the cash drawer of the grain of the conventional alumina ceramic material, and gives the means for the manufacture being easy and mass-producing cheaply the parts of vacuum deposition equipments, such as a gas inspersion shield and other productions, further.
[0046] The vacuum deposition equipment using the parts equipped with the alumina ceramic material of this invention, especially CVD reactor can be used for processing of the material of a silicon wafer or others, and show a pollution reduction of grain.
[0047]
[Effect of the Invention] As explained to the detail above, the polycrystal alumina ceramic material which shows the high resistance over a plasma fluorine by the alumina ceramic material and its manufacture technique of this invention, and shows a reduction of grain occurrence by them is offered.

[Translation done.]

* NOTICES *

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the typical cross section of the CVD system of the conventional technique using the gas inspersion shield which may be equipped with the polycrystal alumina ceramic material.
[Drawing 2] It is the perspective diagram of the status that the lid of the CVD system shown in drawing 1 opened, and is a perspective diagram showing the relation between a gas inspersion shield, a blocker, and a chamber lid.
[Drawing 3] It is the microphotography (scale factor x9000) of a polycrystal alumina ceramic material after sintering for 4 hours.
[Drawing 4] It is a microphotography (scale factor x9000) after sintering for 8 hours to the polycrystal alumina ceramic material which has the same composition as what is shown in drawing 3 , and is the photograph in which it is shown that the total of non-sintered grain decreases according to this invention.
[Description of Notations]
10 -- -- CVD system, 12 -- deposition chamber, 13 -- vacuum channel, and 14 -- -- an evacuation system, 16 -- gas inflow means, 17 -- gas inspersion shield, and 18 -- -- a blocker, 20 -- wafer lift, 22 -- baffle plate, and 24 -- -- a lift finger, 26 -- susceptor lift, 28 -- substrate, and 30 -- -- a susceptor, 32 -- heating means, 34 -- reaction zone, and 36 -- -- a quartz watch window, 38 -- chamber lid, and

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[Translation done.]

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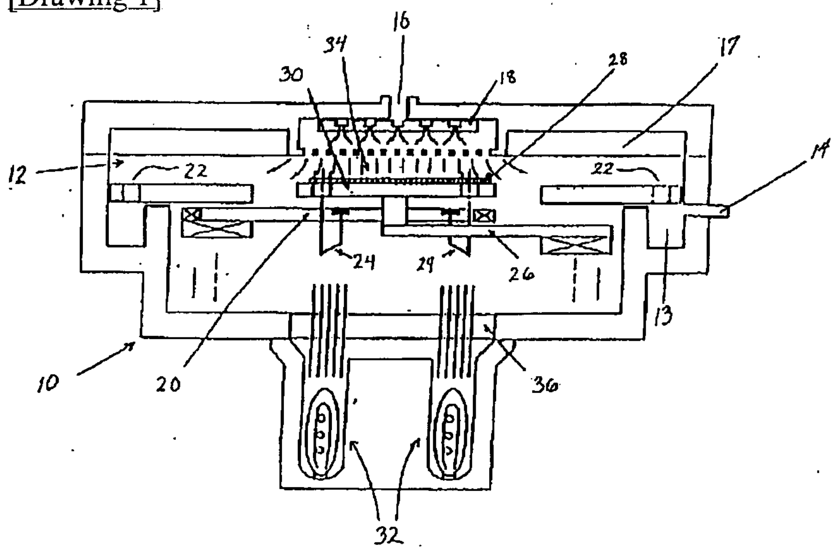
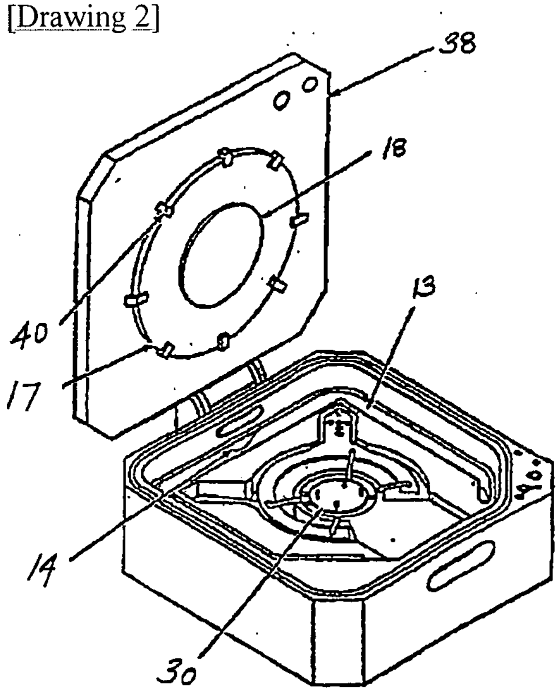
DRAWINGS

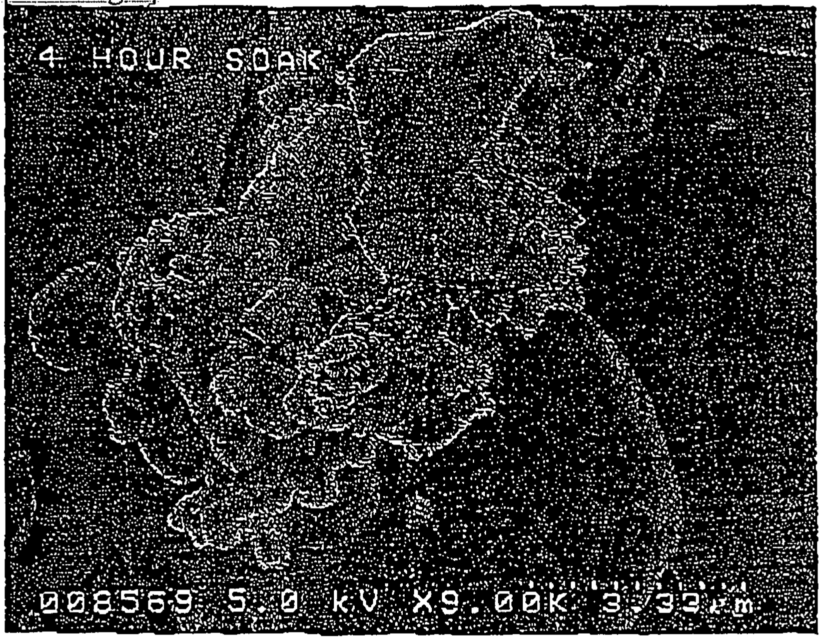
[Drawing 1]



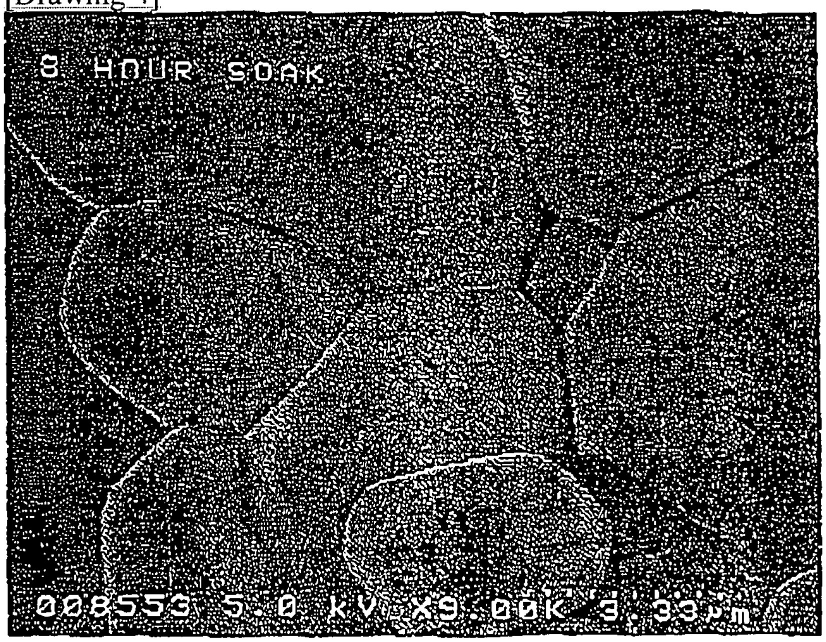
[Drawing 2]



[Drawing 3]



[Drawing 4]



[Translation done.]